Droplet Size Correlation Modeling In Agitated Immiscible Liquid Systems

Hamit Topuz, W.L.Wilkinson

Abstract: This study aims to present experimental studies on the dispersion of immiscible liquids in agitated vessels for a range of liquids and geometries under both batch and continuous conditions. Preliminary experiments were first carried out in a small-scale single-stage mixer-settler unit operating with the 20% TBP/OK, 0.05M nitric acid, uranyl nitrate system. This work was followed by measurements of drop size distribution in agitated liquid-liquid systems in 6 and 9 inches baffled and a 20 cm side square tank using six-flat bladed turbine type impellers. The drop size measurement technique employed fiber optic probes and photography. The liquid-liquid systems used for the measurements were methyl-iso-butyl ketone/water, cyclohexane/water and 20%TBP/OK/0.05 nitric acid. The volumetric fractions were 0.10, 0.15 and 0.20 Impeller speeds varied from 300 to 500 r.p.m. A correlation of the mean drop size for the cylindrical and square vessels under both batch and continuous conditions were established.

Index Terms: Mixer-settler; liquid-liquid dispersion; TBP; drop size; dispersed phase fraction; the Sauter mean.

1 SCOPE AND CONTENT

HE dispersion of immiscible liquids in agitated vessel has been studied for a range of liquids and geometries under both batch and continuous conditions. Preliminary experiments were carried out first in a small-scale single-stage mixer-settler unit operating with the 20% TBP/OK, 0.05M nitric acid, and uranyl-nitrate system. This was followed by measurements of power consumption and drop size distribution in agitated liquid-liquid systems in 6 and 9 inches baffled, and a 20 cm side square tank using six-flat bladed turbine type impellers. The drop size measurement technique employed a rigid fiber optic probe, knit-mesh coalescer aid pad and photography.

2 INTRODUCTION

The growing importance of dispersed systems in numerous industrial and engineering processes led to close examinations on the behavior of such systems, particularly those concerned with solvent extraction processes. Further, In industrial chemical, pharmaceutical, mining, petroleum and food industries, the mixing of two immiscible liquids in turbulent flow is a common operation. Vessels agitated by mechanically in most applications are generally operated under batch conditions, and open to air, more commonly with a rotating impeller mounted on a vertically positioned shaft. Liquids agitated may be partially or completely miscible. Under these considerations if the vessels are not equipped with baffles then the formation of a vortex is inevitable at all, but lowest agitator speeds. One of the aims in producing such dispersions in agitated vessels is to predict the interfacial area which has got a direct influence on the heat and mass transfer operations, and in heterogeneous reactions. The size distribution of the droplets plays an essential role in the overall performance of these processes. In stirred tanks the mass transfer rate between the phases in liquid-liquid systems does not only depend on the dynamics of the motion between the two immiscible liquids.

Much more dependence is given from the particle size distribution of the drops.

3 OBJECTIVES AND BACKGROUND TO THE STUDY

This study is concerned with dispersions of immiscible liquids. The main purpose of such dispersions is to increase the interfacial area so that mass transfers with or without chemical reactions takes place. The equipment employed to produce these dispersions is very varied. They range from mixer-settlers to spray towers. The design of these devices at present is rather empirical due mainly to insufficient studies on dispersion behavior under varying conditions. For instance, although the design of large scale mixer-settlers for uranium purification is usually carried out by simply scaling-up the existing dimensions of a smaller unit. The basis for design is still not understood in spite of many years of operating experiences. The main problem often lies in design of the settler and in order that this may be optimized it is necessary to establish the relationship between settling capacity and the main flow sheet conditions, and the design parameters. It was therefore decided that some limited work on a small scale mixer-settler could be justified with a view to studying the effects some of the main parameters, and to allow the performance of knit-mesh pads as settling aids to be assessed. This work was a precursor to work on the droplet formation. The uranyl nitrate/tri-butyl-phosphate system was used. The primary purpose of this work was to study the drop size distribution of dispersions in agitated vessels. It became clear from the coalescence studies that droplet size had a marked effect on settling behavior. It would be therefore of interest to be able to predict droplet size distribution from the agitated conditions in a mixer settler and then to study the coalescence of the resulting dispersions of immiscible liquids at various dispersed phase holdups. The dispersion of methyl-iso-butyl-ketone in water, cyclohexanol in water and 20/80 by volume tri-butyl-phosphate/odorless kerosene in 0.05M nitric acid dispersions were produced in 6-9 inches in diameter fully baffled cylindrical and 20 cm a side square vessels. The application of direct photography by means of fiber optic probe was to develop. This is a novel technique which makes it possible to measure drop size distribution without seriously affecting the hydro dynamic conditions which had been established.
4 LITERATURE REVIEW

Due to the growing importance of liquid–liquid dispersions, many experimental studies on droplet sizes in stirred tanks coupled with a settler unit have been reported in literature. Rietma [1] made a discussion of control of reaction rate in stirred tanks by means of drop size, and interfacial area. Hinze [2] says that drops are broken up by the pressure caused by the turbulent flow in the vicinity of drop surface. He said that coalescence may occur when drops collide each other. Vermeulen at al. [3] measured interfacial areas in L–L, and gas-liquid dispersions produced in baffled cylindrical tanks by use of light transmission. He correlated his data in the following form

\[ \frac{d_{32}}{L} = C_1 \left(\frac{\nu e}{L}\right)^{0.40} \]  

(1)

Roger et al. [4] did measure average drop size in liquid-liquid dispersion of equal volumes of both liquids. He represented a correlation as

\[ \frac{d_{32}}{L} = C_2 \left(\frac{\nu e}{L}\right)^{0.38} \left(\frac{L}{\nu}\right)^{-0.25} \]  

(2)

Here, \( k \) varies from 0.75 to 1.4 and C2 was found to be depend upon the vessel size. T. Calderbank [5] made a correlation of his data derived from nearly 0 to 20% dispersion phase presence. He correlated his findings as in the form of

\[ \frac{d_{32}}{L} = C_3 \left(\frac{\nu e}{L}\right)^{0.50} \]  

(3)

Where a deviation of was inevitable. This is similar to Vermeulen’s correlation. Shinnar and Church [6] predicted average drop size by means of Kolmogorov’s [7] Theory of Universal Equilibrium. Weinstei...
TABLE 1

PHYSICAL PROPERTIES OF LIQUIDS USED AT 200C

<table>
<thead>
<tr>
<th>Liquid</th>
<th>ρ (g/cc)</th>
<th>μ (cP)</th>
<th>σ (dyne/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20% TBP/OK</td>
<td>0.82</td>
<td>3.00</td>
<td>14</td>
</tr>
<tr>
<td>0.05M HNO3</td>
<td>1.03</td>
<td>1.02</td>
<td>-</td>
</tr>
<tr>
<td>Cyclohexanol</td>
<td>0.96</td>
<td>681</td>
<td>4.500</td>
</tr>
<tr>
<td>Distilled water</td>
<td>1.00</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>MIBK2</td>
<td>0.800</td>
<td>0.57</td>
<td>10.50</td>
</tr>
</tbody>
</table>

(2) Taken from I.C.T
* - Interfacial tension against aqueous phase (0.05M HNO3)

5.2. General description of equipment

The rig consisted of a single–Perspex mixer-settler unit which approximates to a 1:3 scale model of an existing production unit at the B:N.F.L Springfield Works. This is more convenient for the experimental purposes. The system was operated with 20% tri-butyl-phosphate in odorless kerosene technical grade and 0.05M nitric acid containing uranium at concentrations typical of plant conditions. The two-phases were circulated that is mass transfer did not take place in the system as is the case under normal plant conditions. The position of the dispersion band was controlled by a gravity leg system which was constructed on the outlet of the aqueous stream flowing into the aqueous storage tank. Some important dimensions are as follows: 1-the mixed-phase port as arranged by the baffles was 25 mm wide by 60 mm heights 2-solvent overflow weir height: 163 mm 3-aqueous under-flow bridge wide by 15 mm. Organic and aqueous incoming flows brought together through a T-piece just before they were introduced into the mixing compartment of the unit. A light unit was installed to allow visual inspection of the emulsion band characteristics. This arrangement gave excellent illumination which helped significantly in the inspection of the emulsion layer in the settler under all conditions. The liquids processed were approximately 15 liters each, so that the phases at minimum and maximum flow rates planned could have at least 3 and 15 minutes for the separation. i.e. the residence time in the unit. A second type of mixed–phase port arrangement given in Fig. 2(a) and 2(b) was also used at the preliminary stage.

TABLE 2

PARTS LIST FOR FIG.1

<table>
<thead>
<tr>
<th>Item number</th>
<th>Description</th>
<th>Item number</th>
<th>Description</th>
<th>Item number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Mixing vessel</td>
<td>10</td>
<td>Solvent pump</td>
<td>19</td>
<td>Solvent phase layer</td>
</tr>
<tr>
<td>2</td>
<td>Settling department</td>
<td>11</td>
<td>Aqueous pump</td>
<td>20</td>
<td>Aqueous phase layer</td>
</tr>
<tr>
<td>3</td>
<td>Knit-mesh pad</td>
<td>12</td>
<td>Hot water pump</td>
<td>21</td>
<td>Switch board</td>
</tr>
<tr>
<td>4</td>
<td>Motor</td>
<td>13</td>
<td>Electronic tachometer</td>
<td>22</td>
<td>Solvent outlet</td>
</tr>
<tr>
<td>5</td>
<td>Magnetic probe</td>
<td>14</td>
<td>Solvent flow meter</td>
<td>23</td>
<td>Aqueous outlet</td>
</tr>
<tr>
<td>6</td>
<td>A piece of ferrous metal</td>
<td>15</td>
<td>Aqueous flow meter</td>
<td>24</td>
<td>Heating elements</td>
</tr>
<tr>
<td>7</td>
<td>Solvent storage tank</td>
<td>16</td>
<td>Solvent feed valve</td>
<td>25</td>
<td>Dispersion band</td>
</tr>
<tr>
<td>8</td>
<td>Aqueous storage tank</td>
<td>17</td>
<td>Aqueous feed valve</td>
<td>26</td>
<td>Mixed phase port</td>
</tr>
<tr>
<td>9</td>
<td>Temperature controlled bath</td>
<td>18</td>
<td>Impeller</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
In both designs the main concern was to eliminate any disturbances caused by the jetting action from the mixing compartment over the organic and aqueous phases which were already disengaged and the dispersion band formed along the liquid-liquid interface in the settler.3-Flow system: It consisted two main stream, namely solvent and aqueous flow. Stainless steel and hard nylon were used for construction of the flow system because of corrosion reason. 4-Pumping system and flow meters: the pumping system consisted of two stainless steel pumps. For filling and emptying the system a controlled syphoning device was used. Two rotameters of capacity up to 10 liters per minute employed.5-Mixing vessels used in batch process: It comprised two cylindrical tanks with inside diameters of 6 in and 9 in, and a height of 7 in, and 10 in respectively and a square tank of side 20 cm. All made of Perspex. The standard tank configurations as recommended by Rushton [18] et al were followed. The cylindrical vessels were equipped with a set of four baffles equally spaced. The square tank was un baffled.6-Impellers and Motor: The impellers are stainless steel flat-bladed turbines. They were mounted on shafts of various heights. The stirrers were driven by a 1/10 HP totally enclosed motor with a variable speed drive up to 2200 rpm. It was operated in conjunction with an electrical tachometer to control the speed.7-The electronic tachometer: It made by sapphire Electronic Ltd., designed for accurate measurements of linearly and rotary speeds. The speed up to 30,000 rpm can be measured by the tachometer. It has interchangeable magnetic and photoelectric probes.8-The air–bearing disc: It consisted two parts. A top plate can be removable and a stationary bottom plate into which compressed air was admitted. The top plate was lifted by the air supply. Until the top plate turns freely compressed air was supplied. By this method all drag forces were eliminated.9-Photographic System: Direct photography was used throughout for the determination of the drop size distribution. The system is designed in such a way that to enable one to photograph the dispersion at different locations within the vessel with a minimum of interference to the flow regime under both batch and continuous flow conditions. The power input was measured simultaneously. It consisted of mainly two fiber optic units, a light guide and a photographic probe, commercially known as image conduit. They are both made rigid bundles of optical grade fibers. With application of heat they can be given any shape required for the experimental purposes.10-Description of the Photographic probe: A fiber optic conduit consisted of thousands of precisely aligned glass fibers coated with a layer of optically denser glass making individual glass fibers reflective. The image formed at one end and then transmitted through internal reflection to the other face. The probe designed specifically for the purpose of the present work was built from a coherent rigid image conduit, 3 mm diameter composed of 7 x 104 fibers of 10 micron diameter. One of the rigid conduits used as the light guide was bent carefully over a Bunsen burner in the form of U. This arrangement was needed in order to reach to different locations in the vessel as shown in Fig.4. One end of both conduits was submerged into the dispersion in the vessel and positioned face to face. The gap between the immersed ends was varied according to the intensity of dispersion. An intense collimated light is transferred to the immersed face and this made an image of the droplets of the dispersion on the immersed face of the image guide conduit. The created then transmitted to the external face of the conduit upon which a 35 mm Pentax camera was coupled. The camera and the probe was mounted on a stand which made the system adjustable in order to get a clear image of the dispersion as desired. This arrangement provided an ample facility for photography with all vessel configurations.

11. The photographic probe assembly: essentially arrangements are similar to a telescope, but different in buildup. A clear picture given in Fig.3.

12. Liquids used for droplet size measurements: In order to study wide range of impeller Reynolds numbers for various types of two-phase liquid systems with different density and viscosities were used. They were selected on the basis of forming strongly, moderate and slowly coalescing systems respectively in an agitated vessel. The three two phase liquids systems are (i) -Methyl iso-butyl ketone with distilled water, (ii) - 20%v/v TBP/OK with 0.05M nitric acid, (iii) -Cyclohexanol with distilled water. All liquids used were of technical grade. They were mutually saturated before they were used for the purpose of the drop size measurements.
13. Experimental Technique used in the phase disengagement: The procedure was used to provide experimental data for the phase disengagement of liquid-liquid dispersion, with or without the use of the knit mesh pads in the rig which was explained earlier on. The total volume of liquids charged to the unit was about 24 liters. The mixer-settler was first filled with heavier aqueous phase by pumping dilute nitric acid solution at a flow rate until 1/3 of the unit was filled. This was followed by solvent phase was pumped in to the stage to fill the remaining part of the unit. This was the case of organic continuous. Similar procedure was allowed for the case of aqueous continuous operation. After all these preliminary checks the run was started under the desired conditions. A steady state was reached in about 30 minutes in all cases. The parameters considered to be taken: (i) the input flow rates of both phases, (ii) the phase ratio, (iii) the type of dispersion formed, (iv) the temperature, (v) the mixer speed, rpm, (vi) the mixer size, (vii) the position of knit-mesh, (viii) the thickness of knit-mesh packing, (ix) the pressure drop across knit-mesh packing, (x) the entrainment samples of each phase.

14. Procedure for measurements: The following procedure was adopted to obtain data for drop size determinations under batch and continuous flow conditions.

A- Batch process:
The mixing vessel was thoroughly cleaned and was placed upon the turn table, filled with the aqueous phase from the distilled water tank and the organic phase according to the volumetric percentage holdup. Impeller was placed at 1:3 of the vessel diameter from the bottom of the tank. The probe and the light guide were located in the vessel facing each other. Agitation has been started then started at the required impeller speed. After adjusting the two probes pictures could be taken at the location required without any interference with the hydrodynamic equilibrium which had been established. During the picture taking process adjustment could easily be made due to the design as shown in Fig. 4. The intensity of the light was controlled by a switch gear attached on the light box. Volumetric samples were taken using 10 ml syringes to inspect visually if, the dynamic equilibrium had been obtained. This was repeated until a convincing reading was reached. Time changed from 10 minutes to 30 minutes depending on the holdup percentage. Before any measurements were made, the magnification of the optical system including camera had to be determined. The magnification of the optical system was found to be approximately 7X. A minimum of 10 and a maximum of 25 exposures were taken in succession to give a drop population of 300 which is recommended to determine the Sauter-mean drop diameter calculations.

B -Continuous process:
The procedure used in the continuous flow experiments was basically the same as for the batch case, except for a few alterations as follows: (i) more time allowed for the system to reach equilibrium. (ii) The flows of both phases corresponding to batch process hold-up fractions were adjusted to give the same hold-up. (iii) more exposure time was allowed as to get a population of 300 well-defined drops on negatives. The rest of the procedure was exactly the same as in the batch processing.

6 RESULT AND DISCUSSION
A large number of experiments were carried out over a wide range of conditions and in each case the total flow rate was varied and the corresponding emulsion band thickness was measured up to the points at which emulsion left the settler through the solvent and aqueous outlets. The effects of mixed-phase port designs on aqueous emulsion band thickness at 550 r. p. m constant impeller speed and at various distances away from mixing section given at Table 2. In addition, the effects of temperatures on emulsion band thickness at various throughputs also given on Table 3.
TABLE 3

EFFECT OF MIXED-PHASE PORT DESIGN ON EMULSION BAND THICKNESS @ 200 C

<table>
<thead>
<tr>
<th>Solvent flow</th>
<th>Aqueous flow</th>
<th>Mixer speed</th>
<th>Mixer size</th>
<th>Port design</th>
<th>Emulsion type</th>
<th>Band thickness at various distance-15 cm</th>
<th>Band thickness at various distance-30 cm</th>
<th>Band thickness at various distance-45 cm</th>
<th>Band thicken. at various distance-58 cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>550</td>
<td>5</td>
<td>(a)</td>
<td>Aqueous</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
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<tr>
<td>1</td>
<td>2</td>
<td>550</td>
<td>5</td>
<td>(a)</td>
<td>Aqueous</td>
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<td>55</td>
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<td>55</td>
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<tr>
<td>1</td>
<td>3</td>
<td>550</td>
<td>5</td>
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<td>85</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>550</td>
<td>5</td>
<td>(a)</td>
<td>Aqueous</td>
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<td>70</td>
<td>70</td>
<td>70</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>550</td>
<td>5</td>
<td>(a)</td>
<td>Aqueous</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>550</td>
<td>5</td>
<td>(b)</td>
<td>Aqueous</td>
<td>10</td>
<td>5</td>
<td>1-2</td>
<td>1-2</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>550</td>
<td>5</td>
<td>(b)</td>
<td>Aqueous</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
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<td>1</td>
<td>3</td>
<td>550</td>
<td>5</td>
<td>(b)</td>
<td>Aqueous</td>
<td>35</td>
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<td>550</td>
<td>5</td>
<td>(b)</td>
<td>Aqueous</td>
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<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>550</td>
<td>5</td>
<td>(b)</td>
<td>Aqueous</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
</tr>
</tbody>
</table>

TABLE 4

EXPERIMENTAL RESULTS CONCERNING WITH VARIOUS TEMPERATURE EFFECTS ON DISPERSION BAND THICKNESS AT VARIOUS THROUGHPUTS

<table>
<thead>
<tr>
<th>Type of dispersion</th>
<th>Temp. OC</th>
<th>Flow rates of phases, l/min</th>
<th>Phase ratio</th>
<th>Throughput (m3h-1m-2) concerning to band thickness-mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>O/W</td>
<td>20</td>
<td>2:2:1/2</td>
<td>4/5</td>
<td>70</td>
</tr>
<tr>
<td>O/W</td>
<td>25</td>
<td>2:2:1/2</td>
<td>4/5</td>
<td>55</td>
</tr>
<tr>
<td>O/W</td>
<td>30</td>
<td>2:2:1/2</td>
<td>4/5</td>
<td>43</td>
</tr>
<tr>
<td>O/W</td>
<td>35</td>
<td>2:2:1/2</td>
<td>4/5</td>
<td>35</td>
</tr>
<tr>
<td>O/W</td>
<td>40</td>
<td>2:2:1/2</td>
<td>4/5</td>
<td>30</td>
</tr>
<tr>
<td>O/W</td>
<td>20</td>
<td>1:1/2</td>
<td>1:2</td>
<td>40</td>
</tr>
<tr>
<td>O/W</td>
<td>25</td>
<td>1:1/2</td>
<td>1:2</td>
<td>32</td>
</tr>
<tr>
<td>O/W</td>
<td>30</td>
<td>1:1/2</td>
<td>1:2</td>
<td>30</td>
</tr>
<tr>
<td>O/W</td>
<td>35</td>
<td>1:1/2</td>
<td>1:2</td>
<td>30</td>
</tr>
<tr>
<td>O/W</td>
<td>40</td>
<td>1:1/2</td>
<td>1:2</td>
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</tr>
<tr>
<td>O/W</td>
<td>20</td>
<td>1:1:1/2</td>
<td>2/3</td>
<td>63</td>
</tr>
<tr>
<td>O/W</td>
<td>25</td>
<td>1:1:1/2</td>
<td>2/3</td>
<td>50</td>
</tr>
<tr>
<td>O/W</td>
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<td>1:1:1/2</td>
<td>2/3</td>
<td>40</td>
</tr>
<tr>
<td>O/W</td>
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<td>1:1:1/2</td>
<td>2/3</td>
<td>33</td>
</tr>
<tr>
<td>O/W</td>
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<td>1:1:1/2</td>
<td>2/3</td>
<td>28</td>
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<tr>
<td>O/W</td>
<td>20</td>
<td>2:3</td>
<td>2/3</td>
<td>80</td>
</tr>
<tr>
<td>O/W</td>
<td>25</td>
<td>2:3</td>
<td>2/3</td>
<td>63</td>
</tr>
<tr>
<td>O/W</td>
<td>30</td>
<td>2:3</td>
<td>2/3</td>
<td>55</td>
</tr>
<tr>
<td>O/W</td>
<td>35</td>
<td>2:3</td>
<td>2/3</td>
<td>40</td>
</tr>
<tr>
<td>O/W</td>
<td>40</td>
<td>2:3</td>
<td>2/3</td>
<td>35</td>
</tr>
<tr>
<td>W/O</td>
<td>20</td>
<td>1:2</td>
<td>1:2</td>
<td>30</td>
</tr>
<tr>
<td>W/O</td>
<td>25</td>
<td>1:2</td>
<td>1:2</td>
<td>25</td>
</tr>
</tbody>
</table>

The drop size measurements were classified into size groups of 20 µ intervals, depending upon the range of sizes resulting from each particular run. The Sauter-mean diameter, dvs sometimes known as d32 was used in this study. Figures. 5,6,7 show the effect of mixer speeds and phase ratio when organic dispersed on dispersion band thickness at various distance from settling point. Further, Fig. 8 show the effect of mixer speed and emulsion type when aqueous dispersed on emulsion band thickness. Topuz and Wilkinson (26).
Fig. 5. Effect of mixer speed and phase ratio when organic dispersed

Fig. 6. Effect of mixer speed and phase ratio when aquorganic dispersed
The reason for the Sauter –mean diameter to use is because it is the most versatile for the prediction of the interfacial area per unit volume may be determined by measurements were plotted on the normal probability paper in terms of cumulative frequency percentage against the drop size. This method was chosen as to show the variation in between drop size distribution on this type of plot. Drop sizes for continuous operations show substantial variation in size from those for corresponding batch operating conditions. A plot of dvs determined versus dvs (experimental) for both vessels from batch and continuous conditions is given in Fig. 9. A plot of dvs (experimental) versus dvs (calculated) for the square vessel is given in Fig.10.

Fig. 7. Effect of mixer speed and phase ratio when organic dispersed

Fig 8. Effect of mixer speed and phase ratio when aqueous dispersed
Fig. 9. dvs calculated versus dvs experimental for batch systems.

A linear relationship was obtained and also the ratio of dvs for continuous to the dvs for batch findings seem to in agreement with the observation of Rushton (18). Furthermore, the power dissipated in the systems undertaken is virtually independent for the ratio of (L/T) in the range of Reynolds Number that was reached. The Sauter mean diameter dvs also known as d32 by definition, is the diameter of a drop possessing the same ratio of the volume to surface area as the total dispersion. Mi is the number of drops with diameter di total number of drops in the dispersion is given by

\[ m_i = \frac{\pi d_i^3}{6} \]

\[ \hat{m} \]

and, the Sauter-mean diameter is calculated from

\[ d_{32} = \frac{\hat{m}_i d_i^3}{\hat{m}} \]

(6)

For a drop of diameter dvs

\[ V_{\text{volume of drop}} = \frac{\pi}{6} d_v^3 \]

(7)

\[ \text{Surface area of drop} = \pi d_v \]

(8)

If, is total volume of the vessel, then is fractional holdup of dispersed phase in the vessel. Thus, is volume dispersed in the vessel. Total number of drops, diameter dvs in the vessel is given by

\[ \frac{6q_v}{p d_v} \]

(9)

or

\[ a = \frac{6q_v}{d_v} \]

(10)

Hence, the interfacial area per unit volume of the vessel is expressed in terms of dvs shown below.

\[ d_{32} \]

(11)

It has also been pointed out that the Sauter mean diameter is equivalent to the weight harmonic mean of the distribution, i.e.,

\[ d_v = \frac{6q_v}{a} \]

(12)

The normal distribution is usually applied when differences of equal amounts in excess or defect from a mean value are equally likely. The equation representing the normal distribution is

\[ y = \frac{1}{\sqrt{2\pi} \sigma} \exp \left( -\frac{(d - \mu)^2}{2\sigma^2} \right) \]

(13)

The majority of reported studies fitted the normal distribution, e.g. Sprow [12], Chen and Middleman [9], Lee [27], Bouyatiotis and Thornton [14].
7 DROPLET SIZE DISTRIBUTION
The photographs were used to obtain cumulative drop size distributions in order that the effects of the agitator speed, the dispersed phase hold-up, total flow rate, and the phase ratio of the solvent–to–aqueous could be determined. Figures: 11, 12, 13, 14, 15, 16 and 17 show hold up profiles dispersion band in different dispersed phase systems.

**Figure 11.** Structure of emulsion band in the vicinity of the mixer—phase port at 200 C when aqueous dispersed

**Figure 12** Structure of emulsion band at the end of settler when aqueous dispersed at 200 C

**Figure 13** Structure of emulsion band at the end of settler when aqueous dispersed at 200
**Figure 14** Structure of emulsion band in the middle of settler when aqueous dispersed at 200°C

**Figure 15** Structure of emulsion band in the middle of settler at 200°C when organic dispersed

**Figure 16** Structure of emulsion band in the vicinity of the mixed-phase port when organic dispersed at 200°C

**Figure 17** Structure of emulsion band in the middle of settler when organic dispersed at 200°C
Present work did not conform the log-normal distribution. The distributions gave approximately straight lines on normal probability paper which indicate that the data are distributed normally. No significant effects of \((L/T)\) were found on the drop size, but moderately influenced by the agitator speeds and dispersed phase hold-ups. Differences observed in the size distributions between the regions were markedly smaller than would be expected. This may be attributed to the lower coalescence frequency. Their sizes in the vessels were observed to moderately dependent upon the impeller speed. The relationship determined similar to other investigators by means of Kolmogorov's (7) theory of local isotropy, and on the basis of the Weber Number predicted that drop sizes were controlled by the droplet coalescence and not by the drop breakup. Cumulative volume frequency used against drop diameter on normal probability paper resulted a family of straight lines. The correlation derived for fully baffled cylindrical vessels under batch conditions may be represented by the following equation:

\[
\frac{d_{\text{v}}}{L_I} = 0.023 \left(1 + 3.4\sqrt{q}\right)(We)^{0.40}
\]  

(4)

and the correlation for the continuous flow for the square vessel it is given by

\[
\frac{d_{\text{v}}}{L_I} = 0.040\left(1 + 1.6\sqrt{q}\right)(We)^{0.45}
\]  

(5)

A variation in the equation parameter to those available in literature for batch and continuous may be a result of the difference in the vessel operating and vessel contents. Bigger mean drop size obtained in continuous flow as compared with the values for batch operation may chiefly be attributed to the flow effects. Measurements of drop size were made under both batch and continuous flow conditions by use of a novel probe which employed fiber optics and photography in agitated vessels. The photographs produced were used to obtain cumulative drop size distributions in order that the effects of the agitator speed, the dispersed phase hold up, the total flow rate and the phase ratio of solvent to aqueous could be determined. Figures 18, 19, 20 shows typical drop sizes in different liquid systems and at different speeds in impeller region.

Fig. 19. A Typical photograph of drops, System: 0.10 M.I.B.K. dispersion, Agitator speed: 400 rpm, Magnification: 43 X, Photograph taken: in impeller region

Fig. 20. A Typical photograph of drops, System: 20% TBP/OK - 0.05 M HNO3 (Aqu.disp.), Agitator speed: 300 rpm, Magnification: 43 X, Photograph taken: in impeller region
Evidently, as the dispersed phase fraction increased so does the drop size over a range of agitation. By this the combined effects of hold-up of dispersed phase and the coalescing properties among the system studied could be understood. The effects of the hold-up on the drop size variation are not the same at different speeds for all the systems studied. These different effects may be justified on the basis of coalescence and drop breakup processes. Batch processes take place in the impeller region, i.e., break up in the stream emerging from the agitator and to lesser mean in the rest of the bulk flow. Although drop coalescence is going on, it can have a little effect on the local drop diameter since if it results in a bigger drop size predicted on the basis of the critical Weber number it will be instantly followed by break-up. Figures.21, 22 show typical settling profiles of phases at various temperatures when organic phase and aqueous phase dispersed at 200 C.

![Fig. 21 Settling profile of phases at various temperature when organic phase dispersed](image1)

Fig. 22 Settling profile of phases at 20 C when organic phase dispersed

Tavarides and Coulaloglou [10] reported drop size proportional to N\(^{-1.0}\). They stated that the drop size dependency upon the agitator speed was moderate while the coalescence frequency was a strong function of the agitator speed. Mylnek and Resnick [28] reported that if the rate coalescence is very low only a limited number of collisions are said to be effective, therefore the effect of coalescence is negligible. The emulsion band for more than 1-2 millimeters showed that the emulsion band mainly consisted of the two regions of concentration as suggested by Barnea and Mizrahi [29]. The fact that the volumetric hold-up has a different effect on droplet size at different agitator speeds can be better explained by the relative values of the droplet breakage forces and the coalescing forces. Shinnar [6] reported that in a system where drop breakup is controlling the mean drop size, dvs is proportional to N\(^{-1/2}\) and in one in which the drop size is controlled by coalescence, the mean droplet diameter is proportional to N\(^{-0.75}\). Therefore, it is expected that a change in the ratio of the two forces controlling these mechanisms as the agitator speed is varied. If it is to be expected that the drop break up forces have a greater effect compared with the effects of coalescence as the agitator speed is increased.

8 CONCLUSION

Drop size measurements have been performed in aqueous in solvent and solvent in aqueous dispersions in batch and continuous flow in agitated liquid-liquid system as given in above in a 1:3 scale of the existing mixer-settler system in BNFL Works at Springfield in England. The drop size measurement in the present study seemed to be moderately dependent upon the agitator speed. The drop became larger as the impeller speed decreased as expected. For the continuous process the dvs found proportional to the impeller speed N-0.30 Wilkinson and Topuz (26). The reason for the lower exponent may be explained by the fact that the coalescence frequency is more dependent on the impeller speed.
speed than is the drop break up process.

9. ACKNOWLEDGEMENT

We sincerely acknowledge the permission, for the experimental purposes, granted by British Nuclear Fuels Limited, Springfield works during this project under the supervision of Prof. W.L. Wilkinson, The Chairman School of Post-Graduate in The Chemical Engineering Department at University of Bradford, England, The UK. Financial support from The Ministry of Education of The Turkish Government is gratefully acknowledged. † Prof. W.L. Wilkinson is deceased. This publication is in name of his memory.

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Nomenclature

\( \alpha \): a constant, 3.1416  
\( A \): Constant  
\( a \): radius of drop (mm)  
\( \dot{a} \): acceleration  
\( C_1, C_2, C_3 \): constants  
\( D_i \): Impeller size (cm)  
\( D_T \): Tank size (cm)  
\( O/W \): oil to water  
\( W/O \): water to oil  
\( S/A \): solvent to aqueous  
\( L \): impeller blade width  
\( d \): drop size, mm  
\( d_{max} \): maximum drop size, mm  
\( d_{st} \): volume-to-surface, mm  
\( d_{sm} \): Sauter mean drop size, mm  
\( d_m \): drop size in a mono layer, mm  
\( M \): total drop number  
\( N(n) \): impeller speed rpm  
\( \theta \): a sign of function  
\( \theta \): holdup percentage  
\( \mu \): viscosity, cps  
\( \sigma \): interfacial tension dynes/cm  
\( \rho \): density, gr/cm\(^3\)  
\( \iota \): Impeller size  
\( d \): dispersed phase  
\( d_i \): initial drop size  
\( c \): continuous phase  
\( T \): Tank size  
\( g \): gravitational force ,cm/sec\(^2\)  
\( d_0 \): initial drop size  
\( H \): liquid level in tank, cm  
\( n_b \): number of baffle  
\( Y \): Normal probability density function  
\( M_i \): number of drops  
\( V \): volume of tank  
\( s \): standard deviation  

Dimensionless Groups

\( N_{Wb} = \text{Weber Number} = \frac{N^2D_T \dot{a}}{s} \)

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